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ROBERT A. TAFT WATER RESEARCH CENTER

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NUTRIENT REMOVAL FROM WASTE WATER

1970 STATUS

ROBERT A. TAFT WATER RESEARCH CENTER
ADVANCED WASTE TREATMENT RESEARCH LABORATORY
FEDERAL WATER QUALITY ADMINISTRATION
U.S. DEPARTMENT OF THE INTERIOR
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NITROGEN REMOVAL - GENERAL

Municipal wastewaters have nitrogen contents in the 15-25 mg/l range in untreated and primary settled wastes; the nitrogen is divided between organic compounds, which are mostly insoluble, and ammonia. In general, we can depend on conventional biological processes to transform almost all nitrogenous components in wastewater into ammonia and biological sludge. Once this has been accomplished, we can design systems to remove ammonia by air-stripping. Ammonia stripping at high pH in cooling towers following lime treatment is effective but cannot be used during freezing weather and may suffer from serios scale problems.

Under favorable conditions, biological processes may also oxidize ammonia to nitrates by a two-step sequence called nitrification. It would be beneficial if waste treatment plants were required to produce nitrified effluent. Ammonia nitrogen in effluents has several undesirable features:

(1) Ammonia consumes dissolved oxygen in the receiving water;

(2) Ammonia reacts with chlorine to form chloramines which are less effective disinfectants than free chlorine;

(3) Ammonia is toxic to fish life;

(4) Ammonia is corrosive to copper fittings;

(5) Ammonia increases the chlorine demand at waterworks downstream.

A nitrified effluent, free of substantial concentrations of ammonia, offers several advantages:

(1) Nitrates will provide oxygen to sludge beds and prevent the formation of septic odors;

(2) Nitrified effluents are more effectively and efficiently disinfected by chlorine treatment;

(3) A nitrified effluent contains less soluble organic matter than the same effluent before nitrification.

A nitrified effluent is far preferable to one containing substantial ammonia. However, ammonia and nitrate are interchangeable nitrogenous nutrients for green plants and algae, as well as bacteria. If the nitrate level is too high and is helping to stimulate undesirable aquatic growths, the effluent can be further treated by biological action to convert the nitrates to nitrogen gas. This process is called denitrification. The best developed method at this time for control of nitrogen compounds is biological oxidation to nitrates followed by denitrification with the aid of methanol.

Selective ion exchange of ammonia with lime regeneration may be practical but the process is still in the pilot stage. Several other processes are being studied including selective ion exchange of nitrate and chlorination of ammonia to liberate nitrogen gas.

NITROGEN REMOVAL BY BIOLOGICAL SUSPENDED GROWTH REACTORS

Success in providing a high efficiency for nitrogen removal by biological denitrification requires that the biological transformation of ammonia nitrogen to nitrate nitrogen be under good process control. Any reduced nitrogen compounds introduced into the denitrification stage will pass through the process unaltered and impair overall nitrogen removal efficiency.

Complex factors are involved in maintaining nitrification with a conventional activated sludge system. If nitrification occurs at all, it may be due only to an unintentional accident of design. A three sludge variation of the activated sludge process, developed at the Robert A. Taft Water Research Center, greatly simplifies the process control problems associated with maintaining nitrification.

The three sludge system allows management of the separate biological transformations which are necessary for successful denitrification. The three sludge systems are staged in sequence, with flow passing from one stage to the next. The first stage is a high-rate sludge system, the second stage a nitrification sludge system, and the third a denitrification sludge system. The high-rate system handles the bulk of the carbonaceous removal and at this station the waste activated sludge is removed. Thus, the nitrification stage receives a predominantly ammonia nitrogen feed and an enriched culture develops because each sludge system has its own sludge recycle. This process design also has other desirable features. The high rate system protects subsequent nitrification stages from toxic chemicals. Since this is a staged system there can be no direct short circuiting of materials from the influent to the effluent. Temperature effects on the enriched culture of the nitrification stage are not as extreme as with a single sludge system which contains only a marginal population of nitrifying organisms.

Once controlled nitrification has been established, the biological denitrification process can be optimized. The nitrified effluent flows to a slowly stirred anaerobic reactor where methyl alcohol is added in proportion to the nitrate nitrogen concentration. The organisms in this stage use the oxygen component of the nitrate radical to oxidize the organic carbon of methyl alcohol. The end products of this metabolism are elemental inert nitrogen gas and carbon dioxide, which are liberated to the atmosphere.

The stage approach to nitrification has been investigated in work at the Robert A. Taft Water Research Center (1) and in large pilot plant operations at the University of Notre Dame (2) and Manassas, Virginia (3). The

process has also been evaluated on a 1 mgd scale at Hazel Crest, Illinois. A summation of these studies show that biological denitrification is a controllable process if the reaction is forced with an organic supplement, such as methyl alcohol. Total nitrogen in an effluent can be reliably reduced to about 2 mg/l. The cost of the methyl alcohol for 20 mg/l of nitrate nitrogen is estimated to be about 2¢/1000 gallons treated. For more information contact:

Mr. E. F. Barth
U. S. Dept. of the Interior, FWQA
4676 Columbia Parkway
Cincinnati, Ohio 45226

Phone: 513-871-1820

References:

(1) Barth, E. F., et al., "Chemical-Biological Control of Nitrogen and Phosphorus in Wastewater Effluent," Jour. Water Pollution Control Federation, December 1968.

(2) Echelberger, W. F. and Tenny, M. W., "Control of Organic and Eutrophying Pollutants by Combined Chemical and Biological Wastewater Treatment," Division of Water, Air and Waste Chemistry, American Chemical Society, Minneapolis, Minnesota, 1969.

(3) Mulbarger, M. C., et al., "Manassas, Virginia, Adds Nutrient Removal to Waste Treatment," Water and Wastes Engineering, April 1969.

NITROGEN REMOVAL FROM WASTEWATERS BY COLUMN REACTORS

Columnar nitrate reduction represents a second alternative to the suspended growth systems as a means of biochemically reducing the nitrate ion to elemental nitrogen. In a packed column, the cell residence time of the surface bound slime is much greater than the hydraulic detention time. This, combined with a large contact surface and short diffusion distances afforded by small media such as sand, provides an efficient system for rapid denitrification of an applied feed.

Work at the FWQA Lebanon, Ohio Pilot Plant (J. M. Smith, 1970, Unpublished) has shown that the smaller media systems (sand to 3/4 inch diameter stone) are effective when operated downflow at surface loading rates of 7.0 gpm/ft and at actual contact times of 5 to 30 minutes. Daily backwashing is required to relieve pressure drop due to the accumulation of suspended solids in the upper portion of the column. The denitrifying slime is firmly attached to the media surface, and is not removed during the backwash operation. Greater than 90 percent nitrate reduction can be achieved within these columns at contact times of 10 minutes for sand and 30 minutes for the 3/4 inch stone. The effluent normally contains less than 2.0 mg/l of nitrate nitrogen with effluent turbidities less than 3 JTU, indicating little solids contribution from the attached organisms.

Larger media varying in size from 1 inch to 2 inch aggregate have been successfully employed to denitrify agriculture subsurface drainage at Firebaugh, California (Tamblyn, T.A., and Sword, B.R., "The Anaerobic Filter for the Denitrification of Agricultural Subsurface Drainage," 24th Purdue Industrial Waste Conference, 1969.) The larger media permits upflow operation without backwashing at the expense of longer contact times and increased effluent suspended solids. Nitrate reduction of greater than 90 percent were achieved in contact times of 1 hour for the 1 inch aggregate and 2 hours for the 2 inch aggregate at temperatures above 12°C. The 2 inch columns have been operated continuously for over six months on agriculture subsurface drainage without the loss of efficiency or solids accumulation.

As with suspended growth denitrification, methyl alcohol is used as the supplemental organic carbon source of choice for columnar denitrification because of its low cost, biodegradability and ease of handling. Approximately 3 mg of methyl alcohol are required per mg of nitrate nitrogen removed including the requirement for deoxygenating the nitrified feed. The chemical cost for removing 20 mg/l of nitrate nitrogen in the presence of 5 mg/l of dissolved oxygen is estimated to be about 2¢/1000 gallons treated. For additional information, contact:

Mr. John M. Smith
U. S. Department of Interior, FWQA
4676 Columbia Parkway
Cincinnati, Ohio 45226
Phone: 513-871-1820

AMMONIA NITROGEN REMOVAL BY STRIPPING WITH AIR

Ammonia can be removed from a wastewater effluent by raising the pH to convert ammonium ion to dissolved ammonia and then contacting the effluent with a sufficient quantity of ammonia-free air. This physical process is called desorption or, more commonly, "stripping."

If the contacting is done in a packed tower, the pressure drop across the tower is about 1.0 psi or 28 inches of water. Since the volume of air required per unit volume of wastewater effluent is very high, about 400 cubic feet per gallon in a countercurrent operation, the cost for power to overcome even this relatively low pressure drop is prohibitive.

The problem of high power cost was solved by investigators at the South Lake Tahoe Public Utility District (Slechta, A. F. and Culp, G. L., "Water Reclamation Studies at the South Tahoe Public Utility District," Jour. Water Pollution Control Federation, May 1967) who used a slat-filled tower such as is used for cooling water to contact water and air. The pressure drop across such a device is very low, about 1/2 inch of water, so power costs are reduced to reasonable levels. Removal efficiencies as high as 90 percent were obtained in a 24-foot high tower in which wastewater effluent and gas were contacted in a nearly countercurrent fashion. On the basis of this experience, a full-scale stripping tower was constructed at South Lake Tahoe. The tower was designed to remove 90 percent of the ammonia from 3-1/2 MGD of Tahoe's removated wastewater. The air flow is not countercurrent to the liquid but flows across the tower (cross-flow), while the wastewater drips downward through the packing.

Initial operation of Tahoe's stripping tower was in the winter and immediately revealed a limitation of ammonia stripping. When air temperature fell below 0°C, freezing of water occurred at the air inlets, making the tower inoperable. Also, since ammonia solubility is higher in cold water than in warmer water, more air is required to remove it (800 cubic feet per gallon at 0°C). The Tahoe tower was designed for 400 cubic feet per gallon; therefore, removal was much lower than 90%.

Another problem which developed at Tahoe is the formation of scale in the tower. The scale is chiefly calcium carbonate. It forms because the previously lime-treated effluent is supersaturated with respect to calcium carbonate. In the case of the tower at Tahoe, the sludge can be flushed from the tower except from inaccessible areas which cannot be reached with a water jet. A pilot scale ammonia stripping tower at FWQA's Blue Plains, Washington, D. C. Pilot Plant, has had similar scaling problems, except the scale is hard and adheres to the tower fill. The causes of the differences in the nature and amount of scale in various locations has not been resolved. Studies are in progress to see if the scale can be prevented from forming, or if it can be made nonadherent.

The cost of ammonia stripping has been estimated for the South Lake Tahoe facility to be about 2.9¢ per 1000 gallons of wastewater treated. This does not include the cost of the lime and facilities to raise the pH to about 11. These costs have been charged to phosphorus removal because this is the direct objective of the lime addition. If 90% removal of ammonia nitrogen is required even in cold weather, these costs should be increased by about 50% to provide for a higher air-to-water ratio.

Ammonia stripping is feasible when the temperature is above freezing but there is danger of serious fouling by scale. The best approach for minimizing scale and its effects appears to be to use a pH of about 10.5, countercurrent operation rather than cross-flow, and an open fill to allow for easy flushing of accumulated solids. For more information contact:

Dr. J. B. Farrell
U. S. Dept. of the Interior, FWQA
4676 Columbia Parkway
Cincinnati, Ohio 45226
Phone: 513-871-1820

NITROGEN REMOVAL BY CHEMICAL METHODS

A. Removal of Ammonia by Selective Ion Exchange

Conventional water softening ion exchange resins which are selective for calcium and magnesium do a relatively poor job of removing ammonium from dilute solutions. Total deionization by mixed bed ion exchange resins will, of course, remove ammonium ions along with other cations but this process is too costly for wastewater treatment.

Certain zeolites show unusual selectivity for the ammonium ion. A number of these have been investigated by the Atomic Energy Commission because they also show selectivity for cesium and potassium ions. A demonstration project at the Battelle Memorial Institute - Pacific Northwest (Hanford) Laboratories, 1969 (Mercer, B. W., et al., "Ammonia Removal from Secondary Effluents by Selective Ion Exchange," Jour. Water Pollution Control Federation, Research Supplement, February, 1970) showed that certain zeolites, including the naturally occurring mineral clinoptilolite, had a high selectivity for ammonium in natural and wastewaters. A trailer mounted demonstration plant with a capacity of 100,000 gallons per day was built as a cooperative demonstration project between the FWQA and Battelle-Northwest. This trailer is now operated under contract to the FWQA to demonstrate selective ion exchange removal of ammonium ions from solution.

Clarified secondary effluent is passed downward through columns containing clinoptilolite. When a column becomes loaded with ammonia, it is regenerated with limewater containing sodium chloride to speed up the rate of regeneration. The high pH of the limewater converts the

ammonium ion to unionized ammonia gas in solution. The ammonia laden limewater is then pumped through a packed column through which heated air is blown to remove the ammonia.

Pilot studies at Battelle-Northwest indicated a cost approaching 10¢/1000 gallons for their Zeolite method. At Lake Tahoe, where winters are severe and the area is not readily accessible for chemical deliveries, a cost of 15¢/1000 gallons has been estimated for a 7-1/2 mgd plant. More reliable cost estimates will be available at the conclusion of the present contract with Battelle-Northwest.

B. Ion Exchange for Nitrate Removal

Several attempts have been made to develop selective ion-exchange processes for nitrate removal. Dow Chemical Company is presently under contract to FWQA (Contract No. 14-12-808) to develop a nitrate removal process based on the use of a porous solid absorbent containing a nitrate-selective water-immiscible extractant. The process has the advantages of liquid ion-exchange technology and the ease of operation of the granular bed resin systems.

Selective nitrate removal by ion exchange will not be feasible until new resins are synthesized with a high selectivity for nitrate over other anions present in the water. In addition, a suitable process for treating the nitrate laden regenerants must be developed.

C. Chlorination of Ammonia

Ammonia can be oxidized to nitrogen gas by chlorinating to the breakpoint with either chlorine gas or sodium hypochlorite. Four moles of chlorine or hypochlorite per mole of nitrogen gas liberated are required. Hypochlorite is more expensive than chlorine gas, but it is much safer to transport and handle.

Breakpoint chlorination, of course, also disinfects the wastewater as well as oxidizing ammonia. However, the addition of 200-300 mg/l of chloride ion would not be acceptable for many inland water.

Assuming 20 parts of ammonia nitrogen in a secondary effluent, 200 parts of chlorine would be required for breakpoint chlorination. This is equivalent to 1.5 lbs of chlorine per 1000 gallons or about 6¢/1000 gallons. To this must be added the cost of handling the corrosive hydrochloric acid produced. Sodium hypochlorite may cost twice as much as chlorine but associated costs are greatly reduced.

For more information on ammonia removal by selective ion exchange or breakpoint chlorination, contact:

Dr. R. B. Dean
U.S. Dept. of the Interior, FWQA
4676 Columbia Parkway
Cincinnati, Ohio 45226
Phone: 513-871-1820

For additional information on selective nitrate removal, contact Mr. R. A. Dobbs at the same address.

PHOSPHORUS REMOVAL - GENERAL

Phosphorus is considered by many investigators to be the key nutrient in breaking the eutrophication cycle. However, conventional secondary plants are not efficient in phosphorus removal. Phosphorus enters a plant in the highest oxidized form. But, no common biological systems reduce phosphorus; therefore, it cannot be liberated in a gaseous form as nitrogen, carbon, and sulfur are. Removal by biological means, then, is limited to cell metabolic needs and whatever excess phosphorus can be encouraged to be taken by and stored by the cells. The quantity stored above the 1% required for maximum growth is usually classified as "luxury uptake."

A few plants have reported efficient phosphorus uptake on a sustained basis, including the San Antonio Rilling Plant and the Baltimore, Maryland Plant. These results cannot be readily duplicated at other plants by manipulation of operating conditions. We have not learned enough about the phenomenon to take advantage of it. The removal of phosphorus by biological synthesis and "luxury uptake" is not a controllable process at this time.

If we are to reliably remove phosphorus from wastewaters on a sustained basis, we must choose the chemical or the chemical-biological methods. Strict chemical methods precipitate phosphorus either in the primary settler or in a tertiary clarifier. The chemical-biological method employs direct chemical dosing to the aerator of an activated sludge plant. The chemically-bound precipitated phosphorus is removed with the sludge and is not resolubilized during sludge disposal unless the pH is substantially lowered. Effluent phosphorus concentrations of 1-2 mg/l as P can be regularly achieved if the precipitation is accomplished in the primary or secondary portions of the plant. Tertiary lime clarification followed by filtration will lower the concentration to less than 0.5 mg/l.

BIOLOGICAL PHOSPHORUS REMOVAL

The literature indicates that several factors exert an influence on biological phosphorus removal. The rate of aeration and the aeration time have been indicated by most investigators as the most important criteria, the rate of air supply probably being the more critical of the two. Aeration rates in the order of 3 to 7 cfm/gal and detention times of 4 to 6 hours appear to be desirable.

There is some disagreement in the literature with respect to optimum concentration of mixed liquor suspended solids (MLSS). Apparently, increased uptake has been attained at both low and high MLSS from 500 mg/l up to 4300 mg/l. At the San Antonio, Texas treatment plants, the optimum appeared to be 1000 mg/l or slightly higher (1). It was also found that the maximum overall phosphorus removal occurred at organic loadings of 45 to 55 pounds of BOD/day/100 pounds of MLSS under aeration.

It also appears essential from the literature that a dissolved oxygen (DO) level of at least 2 mg/l should be maintained in the last half of the aeration tank to insure that phosphorus will not be released in the secondary clarifier. It is possible that a still higher DO level of 3 to 5 mg/l may be advantageous to maintain a minimum DO concentration of 1.5 mg/l in the sludge until it is through the secondary clarifier. Phosphorus leakage or resolubilization will occur in the secondary clarifier when the sludge consumes available dissolved oxygen. It has been suggested that solids detention time in final clarifiers should be less than 30 minutes.

These key design criteria and operational parameters have not been sufficiently isolated and identified to effectively predict and implement controlled phosphorus removal by the solely metabolic mechanism. As more data has been collected, an alternative chemical explanation has been advanced (2). Simply states this theory indicates, especially in hard water areas, that phosphorus can be precipitated within the biological floc as calcium phosphate at the end of the aeration period, where carbon dioxide is scrubbed from the water by aeration and a substantial increase in pH occurs. This amount of precipitated calcium phosphate and the precipitation of additional phosphorus by traces of iron, aluminum, and magnesium normally present in wastewater would produce an efficient overall removal.

The calcium phosphate theory has been tested at several treatment plants with erratic results. Operating a segment of the Hyperion, California Plant according to the guidelines outlined by the theory has greatly increased the efficiency of phosphorus removal. At Baltimore, Maryland where efficient phosphorus removal occurs routinely, observations show no major increase in pH during operation. Studies at Texas City, Texas where attempts were made to deliberately force calcium phosphate precipitation by the addition of 200 mg/l of lime to the aerator have not shown efficient removal.

The preliminary data reported from these full-scale treatment plants are still not complete or detailed enough at this date to confirm either the metabolic or calcium phosphate precipitation theory. For further information contact:

Dr. C. H. Connell
The University of Texas Medical Branch
Department of Preventive Medicine and
Community Health
Galveston, Texas 77550
or

Dr. R. L. Bunch or Mr. E. F. Barth
U.S. Dept. of the Interior, FWQA
4676 Columbia Parkway
Cincinnati, Ohio 45226
Phone: 513-871-1820

References

(1) Vacker, D., et al., "Phosphate Removal Through Municipal Wastewater Treatment at San Antonio, Texas," Jour. Water Pollution Control Federation, May 1967.

(2) Jenkins, D. and Menar, A. B., "The Fate of Phosphorus on Waste Treatment Processes: the Enhanced Removal of Phosphate by Activated Sludge," Proceedings of the 24th Purdue Industrial Waste Conference, 1969.

PHOSPHORUS REMOVAL BY MINERAL ADDITION TO THE PRIMARY OR SECONDARY

Mineral addition is out of the research stage and into the application stage. Field experience on full-scale and large demonstration pilot plants shows that ferrous, ferric, and aluminum salts can be equally effective as phosphorus precipitants in wastewater. Plants can accomplish 80 to 90 percent phosphorus removal with a minor investment in capital equipment for chemical storage tanks, chemical pumps, and control equipment (Barth, E. F. and Ettinger, M. B., "Mineral Controlled Phosphorus Removal in the Activated Sludge Process," Jour. Water Pollution Control Federation, August,1967).

For trickling filter plants, the chemical precipitation should be accomplished in the primary tank. Direct dosing of chemicals to the trickling filter has not proven highly effective. A small dose of polymer is needed to flocculate and settle the phosphorus which is insolubilized by the mineral addition. Subsequent passage through the trickling filter to satisfy metabolic needs serves as a polishing step. Dow Chemical has conducted several studies of iron-polymer precipitation in the primary at Midland, Lake Odessa, Grayling, and Benton Harbor, Michigan. FWQA sponsored projects include Grand Rapids, Michigan (45 mgd) and Richardson, Texas (1.5 mgd). For further information contact:

Mr. Ronald F. Wukasch
The Dow Chemical Company
2020 Abbott Road Center
Midland, Michigan 48640
Phone: 517-636-2634

With an activated sludge plant, it makes very little difference where the point of addition of the metal ion is. Efficient removals have been obtained when dosing raw wastewater before primary settling, after primary settling, in the aeration tank, or near the mixed liquor exit point. Physical constraints of a particular plant may favor one point of addition over another. However, the key factor in this approach is that no matter where the metal ion insolubilizes the phosphorus, the overall plant efficiency is dependent upon the ability of the biological floc to

collect these dispersed precipitates and remove them from the final plant effluent. Polymer addition in the primary is not necessary for an activated sludge plant as the naturally occurring polymeric materials in the mixed liquor will serve the same purpose. FWQA sponsored projects of phosphorus precipitation in an activated sludge plant include Penn State University (2 mgd), Texas City, Texas (0.75 mgd), University of Notre Dame (50,000 gpd), Manassas, Va. (1 mgd), Xenia, Ohio (1 mgd), and Detroit, Michigan (7,000 g.d). For more information contact:

Mr. E. F. Barth
U. S. Dept. of the Interior, FWQA
4676 Columbia Parkway
Cincinnati, Ohio 45226
Phone: 513-871-1820

Dosages of 1.5 to 2.0, on a molar basis, of metal ion to phosphorus can produce effluents with a residual total phosphorus of 1 milligram per liter or less consistently on full-scale application. As is true with other parameters such as BOD, COD, and suspended solids, if very low residuals are desired, filtration of the effluent would be required. If commercial aluminum and iron minerals are used, the chemical cost will vary from 2-5¢/1000 gallons, depending on the phosphorus concentration and the chemical employed. If waste pickle liquor is available for the cost of trucking only, the chemical cost may be as low as 0.5¢/1000 gallons.

The mineral addition process can be superimposed on the three sludge biological denitrification process to achieve efficient phosphorus, nitrogen, carbon, and suspended solids removals in one integrated treatment sequence (Barth, E. F., et al., "Chemical-Biological Control of Nitrogen and Phosphorus in Wastewater Effluent," Jour. Water Pollution Control Federation, December,1968). In this process, the recommended major point of mineral addition is to the high-rate unit with a small polish dose to the denitrification unit to precipitate residual phosphorus.

LIME PRECIPITATION OF PHOSPHORUS

Dorr-Oliver's Phosphate Extraction Process (PEP) is the only commercially advertised lime precipitation process for use in the primary. In this process, a solids contact type reactor-clarifier is used instead of a conventional primary settler. Clarifier underflow solids are recycled to the raw sewage to maintain 500-2,000 mg/l of suspended solids in the reactor. The objective of the PEP process is to achieve 80% phosphorus removal in the primary, depending on the subsequent activated sludge step to increase overall removal to 90% or greater. For further information contact:

Mr. O. E. Albertson
Dorr-Oliver Incorporated
International Headquarters
Stamford, Conn. 06904
Phone: 203-348-5871

Currently, lime precipitation is also being considered as the first step in a chemical-physical treatment sequence for raw wastewater that does not include a biological unit. Subsequent units in the sequence include lime recovery, filtration, carbon adsorption and possibly ammonia stripping.

The above sequence is similar to the tertiary sequence demonstrated for several years at Lake Tahoe's 7.5 mgd Water Reclamation Plant. The Lake Tahoe Plant utilizes secondary effluent as feed water. Phosphorus removal costs at Tahoe vary monthly from 6-8¢/1000 gal. including amortization, operating costs, and recalcination.

For additional information on chemical-physical treatment of raw wastewater, contact:

Mr. J. M. Cohen
U. S. Department of Interior, FWQA
4676 Columbia Parkway
Cincinnati, Ohio 45226
Phone: 513-871-1820

For additional information relative to Lake Tahoe's operations, contact Dr. R. B. Dean at the same address.